Abstract

Over the past 3500 years, niello has been utilized by almost every culture with a tradition of decorative metal work. However, it has been abandoned by modern western jewellers. Traditionally niello is manufactured using lead, making it unsafe, and unsuitable for use in modern jewellery. This paper will explore the historical manufacturing and use of niello to provide context, as well as present how to manufacture a lead-free version for safe use by modern jewellers.

Niello 2.0

Christopher M. Manning Silver Hand Studios, Ottawa, Canada

Niello was an important alloy used by metal-smiths from ancient times until the end of the Renaissance. The low-temperature metal sulphide was commonly used as a high-contrast, fusion inlay alloy. The colour permeates the alloy and ranges from dark grey to deep black. It is an excellent choice when making high-wear items where patinas and other surface treatments will wear. It was regularly fused into bronze, iron, silver and gold.

My first introduction to niello came from Cellini's *Treatise on Goldsmithing and Sculpture*¹. Since first experimenting with niello seven years ago, it has become an important material in my art.

Niello was an important alloy in Renaissance Italy and commanded the first chapter in Cellini's Treatise. While the Renaissance was the pinnacle of niello production, its use in decorative metalwork spans from the ancient Egyptians and Romans up to modern-day Russian and Thai jewellers. The Fuller Brooch, located in the British Museum, is one of the finest surviving examples of niello work (Figure 1).



Figure 1- The Fuller Brooch - Courtesy of The British Museum Late ninth century Anglo-Saxon brooch made of silver and niello

Over the past seven years I have explored some of the most common historical recipes of niello. While many traditional recipes withstand the test of time, there are significant toxicity and contamination issues associated with their use. Most traditional recipes include lead to lower the melting temperature. This paper will discuss the history of traditional niello alloys, as well as provide a modern alternative using tin as a lead replacement.

History

Various forms of niello have been used in decorative metalwork for thousands of years. Early recipes used silver and copper alloys and were applied primarily to bronze. The early lead-free alloys are difficult to work with. They were primarily silver and copper, which require high melting temperatures for application. The high temperatures can cause the sulphides to break down and can also come close to the melting temperature of the metal into which the niello is being inlaid. By the early Middle Ages, lead was added to the silver/copper alloy to lower the melting temperature. Niello's use on bronze decreased, while its use on silver, gold and iron increased. A number of early niello recipes have survived, including recipes by Pliny the Elder, Benvenuto Cellini and Theophilus.

The earliest written reference to a recipe for niello appears in book 33 of Pliny the Elder's *Natural History*². He makes reference to ancient Egyptians staining silver with sulphur, although it looks like this is a process of blackening silver using liver of sulphur. The recipe he provides for niello contains silver and copper but lacks lead, making it difficult to work with compared to more modern recipes with a lower melting temperature. As the higher melting temperature would break down the sulphides, it would be applied at a lower temperature as a hot, soft paste. The Romans were fond of using niello on bronze. The higher melting temperature of the bronze made it easier to work with the higher-temperature niello.

Sometime in the early Middle Ages, the addition of lead to reduce niello's melting temperature gained popularity. By the time niello is discussed in *Mappae Clavicul*³ in AD 600, lead is a staple in all recipes and will remain so. Its addition sufficiently lowers the melting temperature of niello to where it can be applied as a liquid without breaking down the sulphides. The twelfth century German monk Theophilus Presbyter discusses the preparation and two methods of application in *On Divers Arts*⁴. The discussion centres around making a chalice, and the application methods are virtually unchanged from what I use today.

While niello recipes have varied dramatically in their proportions, the principle ingredients have not changed. Unfortunately, the lead content in traditional niello makes it dangerous for both artists and consumer. Modern consumer protection laws in many countries prohibit lead in jewellery, which makes traditional niello recipes inappropriate for this purpose. Lead also presents a significant long-term health hazard to artists who prepare and use niello, and it is also a contamination hazard in the shop. A modern alloy of niello is needed for it to be used safely in the modern shop. Despite tin being a well-known metal earlier than the first examples of lead-based niello, there is no evidence of tin being used as an alternative to lead.

The Table 1 includes all of the recipes discussed in the paper, including historical recipes found and tested as part of my early experimentation. The lead-based recipes are provided here for reference, and historical context. I do not recommend using these recipes on modern work unless there is a specific reason to use a traditional recipe. All but one recipe contains lead and should be used only with extreme care.

Table 1

| Source | Silver | Copper | Lead | Tin |
|-----------------|--------|--------|------|-----|
| Pliny | 3 | 2 | 0 | 0 |
| Theophilus | 4 | 2 | 1 | 0 |
| Cellini | 1 | 2 | 3 | 0 |
| Fike | 3 | 1 | 1 | 0 |
| "Modern French" | 3 | 7 | 5 | 0 |
| Wilson | 6 | 2 | 1 | 0 |
| Manning | 7 | 3 | 0 | 5 |

Pliny (3:2:0:0)

The first recipe stands out from the rest as it is the only historical one which is lead free. It is a pre-medieval recipe and would have been used primarily on bronze. The niello produced is a deep, consistent black. The lack of lead changes it in a few ways from the other recipes. First, it requires a very high temperature to apply. The temperature is high enough to cause the sulphides to break down. This recipe is also very hard and brittle. Scraping was not possible without shattering the niello, and it was substantially harder while filing than the other recipes. Other than its lack of lead, this recipe has no advantage over the others. I would not recommend using it.

Theophilus (4:2:1:0)

This is the first of the two medieval recipes tested. It is well documented in Theophilus' treatise, *On Divers Arts*. It increases the proportion of silver as well as adding a small amount of lead, unlike Pliny. The addition of lead decreases the melting temperature slightly and improves the workability. It fills both large areas and fine details easily. Excess niello can be easily scraped or filed away. Much of my past work has relied upon this recipe, and it is recommended.

Cellini (1:2:3:0)

The third recipe comes from Benvenuto Cellini's *Treatises on Goldsmithing and Sculpture*, published in the fifteenth century. Niello work was extremely important to a goldsmith's livelihood in Renaissance Italy, and Cellini discusses its use in the first chapter of his treatise. His recipe opts for a large proportion of lead and small proportion of silver. I suspect this choice was to save precious metal and reduce cost. Application was very challenging. It required significant heat to allow it to flow into all areas of detail and had difficulty filling

consistently. The most significant difficulty comes from the silver precipitating out of the solution. It creates areas of silver instead of the dark grey of the rest of the niello, and it obliterates many details. I would not recommend using this recipe.

Fike (3:1:1:0)

This recipe comes from Phillip Fike's paper published as part of *Metals Technic*⁵. It was very easy to work with and flowed into all areas of the work without difficulty. Clean-up using a scraper or file was easy. The result is not as black as with Theophilus' recipe, and some grey mottling is visible where the silver has partially come out of solution. The mottling is not as pronounced as with Cellini and is not apparent from arm's length. It is an acceptable recipe.

"Modern French" (3:7:5:0)

The "Modern French" recipe comes from H. Wilson's *Silverwork and Jewellery*⁶ from the turn of the last century. It contains the largest percentage of copper as well as a large percentage of lead. It produces the best black of any of the six recipes. The black is very consistent and very deep. It was easy to apply, and clean-up was simple. It is closest to Theophilus' recipe in terms of flow and clean-up. I would recommend this recipe and would be my choice if I needed a lead-based recipe.

Wilson (6:2:1:0)

The final recipe is very similar to Theophilus'; however, it contains a larger percentage of silver. It is Wilson's preferred recipe, but the results are not as consistent as with Theophilus. It fills well and maintains details. The mottling is slightly more pronounced than Fike, and overall it is not as black as Theophilus or "Modern French." The mottling is visible at arm's length if you know what to look for. It is not recommended.

Searching for a Modern Recipe

As discussed, traditionally there are four elements used in making niello: silver, copper, lead and sulphur. Increasing the silver content raises the value of the niello, lowers the melting temperature by a small amount, and lightens the colour. Increasing the copper content raises the melting temperature and makes the niello blacker. Increasing the lead content significantly lowers the melting temperature. Introducing sulphur to the molten alloy converts it into a metal sulphide, causing it to turn black.

Reducing the melting temperature of niello is crucial. Without reducing the melting temperature of the silver/copper-only alloy, it becomes challenging to apply, and the sulphides begin to break down.

In an attempt to find a replacement for lead, I examined other elements surrounding it on the periodic table for their suitability. There were three primary criteria for selection: low melting temperature, boiling temperature above the melting temperature of the silver/copper alloy and low toxicity. The following metals were tested as replacements for lead (Figure 2):



Figure 2 – The raw materials used during the experiment

Gallium

Gallium (Ga) is number 31 on the periodic table. It appears between zinc and germanium, aluminium and indium. It has a melting temperature of 30°C, and a boiling temperature of 2400°C. Gallium is non-toxic. It is liquid near room temperature, which can make it difficult to measure and handle. Gallium is relatively easy to obtain and has a higher cost relative to the other alternative metals.

Gallium was quickly discounted as a substitute for lead. Despite repeated applications of sulphur, the niello never turned black.

Indium

Indium (In) is number 49 on the periodic table. It appears between cadmium and tin, gallium and thallium. It has a melting temperature of 156°C and a boiling temperature of 2072°C. Indium is mildly toxic if injected into the blood stream. It is not absorbed through the skin and is not well absorbed by ingestion or inhalation. It is a very ductile metal, which makes it very easy to work with in the shop. It can be easily cut with a knife or scissors. Indium is easy to obtain and has a higher cost relative to the other alternative metals.

Indium did not reduce the melting temperature of the niello low enough to be a viable alternative. It does not flow well until it reaches over 500°C. As a result, there is a high risk

of the sulphides breaking down. On its own, indium is unsuitable as an alternative in niello.

Tin

Tin (Sn) is number 50 on the periodic table. It appears between indium and antimony, germanium and lead. It has a melting temperature of 232°C and a boiling temperature of 2602°C. Tin does not represent a health risk. It is very ductile and when purchased as sheet, it is easy to cut with scissors. Tin is very easy to obtain and is relatively inexpensive.

Tin proved to be the best alternative tested. It flowed well starting at 230°C, and easily filled both large and small areas in the pattern. The colour was dark and consistent. The section below on making niello will use a tin based recipe.

Lead

Lead (Pb) is number 82 on the periodic table. It appears between thallium and bismuth, tin and flerovium. It has a melting temperature of 327°C and a boiling temperature of 1749°C. Lead does represent a long-term health risk. The primary risk of exposure is through inhalation and ingestion. Exposure is gradual and symptoms are often not obvious until a significant build-up occurs. Because of the dangers of lead exposure, a replacement is needed.

Bismuth

Bismuth (Bi) is number 83 on the periodic table. It appears between lead and polonium, antimony and moscovium. It has a melting temperature of 271°C and a boiling temperature of 1564°C. Bismuth has low toxicity and because it shares many physical properties with lead, it has become a common replacement for lead. Bismuth is used in some cosmetics and medicine, and it does not present a significant risk for use in the studio. It is often found in a crystal form and is very brittle. It is easiest to smash into smaller pieces when measuring. Bismuth is easy to obtain and relatively inexpensive.

Bismuth created the lowest temperature niello of the four tested. It flowed at 200°C and was the closest in terms of flow and fill qualities to traditional, lead-based niello. During testing I was unable to achieve a consistent colour. The silver would precipitate out of the alloy causing large areas of silver coloured metal where it should be black. With further experimentation it may be possible to achieve an acceptable application of bismuth-based niello, however, I did not pursue it in my experiments.

Making Niello

Manning (7:3:0:5)

The recipe presented in this paper was selected with the intention of reducing the long-term health risks to the artists using them. While the elimination of lead from the alloy has removed the most significant health risk, there are still important steps required to work safely with niello.

During the preparation of niello, all normal precautions should be taken while working with torches, high heat, and molten metal. The most important safety consideration compared to normal alloying procedures is the sulphur dioxide produced from burning sulphur in air. Sulphur dioxide readily reacts with the moisture in mucous membranes and forms sulphurous acid, a severe irritant. Mild exposure will result in airway restriction, coughing, sneezing and eye irritation. Higher exposure will result in bronchospasms, resulting in an obstructed airway and severe eye irritation.

Sulphur dioxide is heavier than air and will pool in low-lying areas of the shop. Good ventilation is important while preparing niello. If adequate ventilation is not possible in the studio, preparation outside is encouraged. Along with adequate ventilation, a full-face respirator should be worn during preparation. For information on how to build an effective fume hood for preparing niello, see Whit Slemmons' excellent article/tutorial entitled, "Studio Ventilation Tutorial."⁷

Care should also be taken when filing, sanding and polishing niello. Dust can present a health risk regardless of the toxicity of the metals. The dust and particles from niello can also contaminate other work. Pitting can occur during high-temperature operations such as soldering with pieces contaminated by niello particles. Care should be taken to avoid cross contamination with other work.

All recipes are prepared essentially the same way, requiring similar equipment and precautions. The silver and copper are first alloyed together. The third metal is added and allowed to mix. The molten alloy is then poured into the sulphur. At that point the niello can be cast into a rod for easy use.

Below is a list of the tools and materials required to perform the above experiments.

- Full mask respirator
- P100 filter for respirator
- Two crucibles
- Graphite stir rod
- Tin
- Scale
- Torch
- · Steel angle iron

- Fume hood
- Sulphur
- Copper
- Silver
- Wire mould
- Borax
- Steel spoon
- Ingot mould

A good fume hood is an absolute requirement for making niello in a shop. The hood must exhaust out of the room, and care must be taken when selecting where the exhaust exits the structure. Be sure to test the draw of your fume hood before you start burning sulphur because the fumes from this process will contain large amounts of sulphur dioxide. Niello can also be made outdoors, as long as there is a reasonable breeze to carry away the fumes (be sure to prepare away from bystanders who are unaware and/or unprotected).

No matter where it is made, everyone in the studio or area must wear an appropriate full-face respirator. A half-face respirator is inadequate for this work as it will not protect the eyes from the sulphur dioxide. The respirator must fit properly and should be fitted with a P100

cartridge. If you are unsure about the respirator or cartridge you currently have, consult with a reputable safety equipment supply house. They will be able to provide you with the correct products to protect against sulphur dioxide.

Wear clothes you are willing to discard. The smell of burning sulphur is invasive, and you should be willing to throw out the clothes you are wearing. Be aware of the dust created when filing, sanding, and polishing niello, as it can also contaminate clothing. The dust can cause issues with other work in the shop.

The sulphur must be in a powdered form. It is unnecessary to use chemically pure sulphur. Garden centres are a good source of sulphur, which is sold as a slug deterrent and is typically 92% pure. Despite some historical sources that discuss specific quantities of sulphur, it is difficult to recommend an exact amount. The goal is to saturate the alloy with sulphur. More sulphur is better, and with experience you will get a sense of how much to work with at a time.

The silver should be pure or contain nothing except copper. If it is an alloy such as sterling or coin silver with a known amount of copper, take into account the copper content when weighing all the metals. The silver should be cut into small pieces or used in grain form to help it melt rapidly.

The copper should be pure. Unused copper plumbing tube is a reasonable source. Even though it often contains a very small amount of deoxidiser, I have not found it to affect the quality of the niello produced. However, do not use pipe taken from an existing plumbing installation as it may be contaminated with solder. Electrical wire can also be used; however, removing the insulation can be tedious. As with the silver, it should be cut into small pieces or used in grain form.

Pure tin can be purchased in several forms. Sheet tin is the easiest to use—a 1-mm thick sheet can be cut easily with scissors.

Preparation is important. Before starting, lay out everything you need so it is easily at hand (Figure 3). Weigh all metals and keep them separate. For tin-based niello, use a recipe of 7 parts silver, 3 parts copper, 5 parts tin. Making 20 grams of niello at a time is reasonable. Disposable paper cups are useful to keep each of the metals sorted.



Figure 3 – Fume hood prepared to make niello

Prepare the two crucibles by coating them in borax and firing them. The crucible used for alloying the metals should be pre-heated before adding any metals. The crucible with the sulphur should not be pre-heated. Fill the sulphur crucible with a spoonful of sulphur.

The steel angle iron should be a few feet long and tilted at a slight angle. You will be pouring the molten niello into it and making a long rod of niello. It should be readily accessible and stable while handling the torch and the crucible of molten niello. Prepare the angle iron by coating it with a thin layer of oil and burning it off to create a layer of soot. There is no need to heat the angle iron prior to pouring the rod.

Once everything is laid out and ready, begin by melting the silver with a reducing flame. When the silver is liquid, slowly add the copper. Stir the alloy as necessary to ensure that everything has melted and mixed. When the silver/copper alloy is liquid, slowly add the tin. It is important to ensure the tin has melted and successfully alloyed with the existing metal before pouring it into the sulphur. Roll the molten metal in the crucible to ensure it is all molten and consistent. Temperature is important from this point on, as high temperatures can damage the sulphides you will create in the next step. Overheating the niello can also cause the silver to precipitate from the alloy.

When you are satisfied the alloy has mixed, pour it into the crucible with the sulphur. Stir the mixture and add heat occasionally until all of the sulphur has been absorbed or burned off. Apply short bursts of heat from the torch to keep the niello molten. While allowing the sulphur to burn off, there may be a small amount of cinder on the molten niello. Do not heat the mixture to the point where the cinder is absorbed into the niello. Instead, remove the cinder with the graphite rod. Once the sulphur is absorbed or burned off, slowly pour it into the angle iron to form a long, irregular rod. The rod should be consistent in colour throughout

and should be brittle. If there is a silver core in the rod, you have overheated the niello or the niello did not absorb enough sulphur. It is impossible for the niello to absorb too much sulphur. Melt the rod and pour it into another crucible of sulphur. Pour a new rod in the angle iron and check it for consistency. Repeat until you have created a consistent rod of black niello.

When you are satisfied with the niello, make it into rods for easy application. A simple wire mould works well for making niello rods for either immediate or later use. Be careful not to overheat the niello when casting into rods or ingots. I would recommend dedicating a wire mould to niello to avoid contaminating future wire of precious metal.

Applying Niello

Using niello in a piece requires planning and preparation similar to using enamel. All soldering operations must be complete before applying niello. At soldering temperatures, the niello will flow again and the high temperature will cause damage to the sulphides. Niello will also cause significant pitting in silver and gold at soldering temperatures. Even extra easy solder requires too high a temperature for use with niello that has already been applied to a piece. Laser or fusion welding is required to repair a piece after the niello has been applied. All stone setting should wait until after the niello has been applied and finished.

A basic finish should be completed prior to application. I recommend finishing with at least P800-grit sandpaper before application. It is best if the area where niello is being applied is flat, or convex, and is easily accessible with files and sanding sticks. All forming should be complete before applying the niello. Do not apply to a flat piece of metal and then try to form it.

Niello is opaque once it is more than 0.05 mm thick. It can be applied into deep or shallow pockets, or engravings. Be cautious about using very shallow depressions, as it is easy to damage the surrounding piece when sanding and possibly destroy the pattern in the process. I recommend making depressions 0.5 mm deep.

If a flux is required to protect the base piece, use a mixture of alcohol and boric acid. Apply the mixture to the work before applying the niello. Heat the piece until the flux has become liquid. The work is now hotter than the melting temperature of the niello. It can be fed by rod through the flux, displacing it. Depending on the size of the piece, it may be necessary to apply heat to the piece to keep it hot enough to melt the niello. Avoid applying heat directly to the niello.

If flux is not required to protect the base piece, apply heat to the base piece and allow it to melt the niello. Once again, avoid applying heat directly to the niello.

The area with niello should be overfilled. There is often damage to the top surface of niello, and overfilling will ensure it can be filed and made level with the surrounding surface. If necessary, use a solder pick to pull niello into all areas of the pattern and prevent air pockets. In particular, pay attention to corners and areas of fine detail.

When filed flat, a second application may be necessary to fill air pockets or areas without

enough niello to reach the same level as the base piece. Slowly heat the piece and watch for the existing niello to melt. Use a solder pick to draw the niello into any air pockets and apply a small amount of fresh niello to overfill the area.

With practice, most pieces can be filled in one or two applications. Be cautious when reheating the piece. It is easy to become impatient and damage the niello with too much heat.

Once applied, niello can be drilled, allowing mechanical fastening of stone settings or other parts. I also commonly use Loctite[®] to secure parts together where appropriate. Low- and medium-temperature Loctite[®] will provide a semi-permanent bond and can be released with heat below the temperature at which niello will melt.

Finishing Niello

There are a few challenges involved with finishing niello. The first challenge is contamination. Any tools (files in particular), sandpaper, buffing wheels, etc. need to be dedicated to niello work. Once a tool has been used for niello work, do not use it on a piece not intended for niello. Also, do not use these tools on a piece until you are ready to work the niello. Due to the tin in niello, if a small particle is on your workpiece while soldering, the high temperatures will allow the tin to alloy with the base piece and cause pits.

You must be careful when finishing niello. It is hard enough to file, sand, and polish; however, it is still softer than the surrounding silver or gold (Figure 4).



Figure 4 - Jaipur pen by author in Argentium® silver and niello

Begin with files and scraper and move to a P220-grit sandpaper depending on the cut of your files. Move through P400-, P800-, P1200-, P1500-, and P2000-grit sandpaper. Be sure to use a sanding stick and add a piece of thick sole leather if you need a slightly more forgiving surface for a three-dimensional surface. Avoid removing too much niello in any one area as it is easy to damage the pattern by being too aggressive in one area. As the underlying silver or gold becomes visible, switch to less aggressive methods for removing the niello.

Niello is hard enough to allow buffing; however, care must be taken not to use an aggressive compound or work a single spot for too long. It is critical that all scratches are removed with sanding before buffing. The niello will be removed faster than the surrounding silver, so use a light touch. Most of the polishing work should be done with paper. Buffing is just the final touch. There is no need to polish beyond a Tripoli.

Niello is valuable, and the filings can be collected and re-melted. You may have some of the base metal mixed in; however, it should be low enough in volume not to matter. I re-melt my filings with a larger mass of niello to avoid issues.

Conclusion

Niello is underused by modern Western jewellers. With a bit of experimentation, there is great potential when combined with 3D printing, CNC machining, and modern engraving to give new life to this ancient technique. With tin based niello it is possible to work safely, provide a safe product for your customers, and create unique work.

"Oh thou discreetest of readers, marvel not that I have given so much time in writing about all this, but know I have not even said half of what is needed in this same art, the which in very truth would engage a man's whole energies, and make him practice no other art at all."

– Benvenuto Cellini

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